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COMBINED USE OF SOLID AND LIQUID FOAMING AGENTS FOR MAKING CELLULAR GLASS AGGREGATE-MICROWAVE ASSISTED

BY

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Abstract. A cellular glass aggregate with excellent heat insulation properties and mechanical strength was designed and tested under conditions of using unconventional microwave heating method. Sodium carbonate and glycerol were utilized as simultaneous expanding agents. Green post-consumer drinking glass was the predominant raw material in the mixture that contained also kaolin and water glass. The optimal cellular glass product had bulk density of $0.22 \text{ g}\cdot\text{cm}^{-3}$, heat conductivity of $0.062 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, and compression strength of 6.9 MPa. The process energy efficiency was excellent, the specific energy consumption being of $0.86 \text{ kWh}\cdot\text{kg}^{-1}$. The originality of the work consists in choosing sodium carbonate as a foaming agent together with glycerol and applying the own technique of mixed heating (direct and indirect) with microwaves.

Keywords: cellular glass, microwave, glycerol, sodium carbonate, water glass.

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1. Introduction

The last decades of the 20th century were characterized by intense activation of recycling reusable materials (metal, glass, plastic, etc.) under much less expensive conditions compared to the price of manufacturing them in their initial state and much fewer emissions of polluting products (Waste and Recycling, 2024).

Glass, the manufacture of which requires a very high energy consumption at high temperatures and considerable emissions of gases with negative effect on environmental protection, is one of materials generating silicate waste with an annual growth rate in continuous expansion. On the one hand, post-consumer drinking glass represents a huge source of such residual products (Scarinci *et al.*, 2005), while the demolition or modernization of buildings is a large supplier of flat glass waste (windows) (Tihomirovs *et al.*, 2023). It has been found in recent decades that finely ground glass waste mixed with an inorganic or organic expanding agent, subjected together to the thermal sintering process causes the formation of glass froth with excellent physical, thermal, mechanical properties, high durability, non-deformability, acid resistance, etc., very attractive for the construction materials industry (Scarinci *et al.*, 2005).

A special product made by expanding the glass waste is cellular glass aggregate (or cellular glass gravel). Its production was the focus of the current work. It is generally obtained in the form of lumps with dimensions between 10-80 mm detached from the sintered, foamed, and cooled mass from the metal conveyor belt of the tunnel kiln at its cold end, aided through the rapid cooling of the material.

According to (Zegowitz, 2015), cellular glass aggregates, that in present are industrially made in tunnel kiln, have several applications in insulation and drainage works. The aggregates are adequate for providing the insulation under concrete reinforced earth slabs, insulation of the perimeter of building's exterior walls, insulation of terraces, industrial roofs, insulation filling in road construction, soil and slope stabilization, insulation of sports fields, insulation of swimming pools and ice rinks, drainage in structural and civil engineering. Some property ranges of cellular aggregate lumps are also mentioned in this work: bulk density (for uncompressed material) between 0.12-0.19 g·cm⁻³, bulk density (for compressed material) within the limits of 0.156-0.247 g·cm⁻³, moisture content between 1-6 vol. %, moisture after 28 days under water in the range of 7-15 vol. %, and heat conductivity between 0.08-0.095 W·m⁻¹·K⁻¹.

Several companies specialize in the production of cellular glass aggregate. Among them, Geocell Schaumglas (Austria), Misapor Switzerland (Switzerland), and Glapor Werk Mitterteich (Germany) are the most well-known in the world. In the Nordic countries of Europe (Scandinavian Peninsula), the cellular glass aggregate market is focused on road construction applications, being influenced by requirements imposed by specific climatic conditions. The

main companies in the Nordic area are Hasopor (Sweden), Glasopor (Norway), and Foamit (Finland) (Cosmulescu *et al.*, 2020).

One of the world's leading manufacturers of cellular glass aggregate, Geocell Schaumglas GmbH, has four production facilities in Austria and Germany. Due to its extremely versatile features (light weight, load-bearing, draining, and insulating) the Geocell type glass aggregate is suitable for using in insulating of basements under slabs, insulating the rooftops, lightweight fill material for landscaping, roof gardens, green roofs, road construction, bridge abutments, insulating underground pipelines, etc. In accordance with (Declaration of Performance, 2016), the glass aggregate has loose bulk density between 0.13-0.17 g·cm⁻³, lump bulk density between 0.22-0.30 g·cm⁻³, lump size in the range of 10-60 mm, water absorption within the limits of 6-9 vol. %, and resistance to crushing over 0.5 MPa.

90% from the glass waste used in the manufacturing recipe comes from post-consumer bottles and other coloured glass and 10% comes from flat glass (mainly from windows). The glass powder is mixed with a natural expanding agent and water glass. The sintering and expanding process occurs at almost 900°C on the conveyor belt of the tunnel kiln. In the cooling chamber zone, the product is fractured due to created internal stresses (Recycled Glass, 2016).

The cellular glass aggregate production of Misapor AG Company takes place in two facilities in Switzerland, one in Germany, and one in Italy. The manufacturing recipe includes 98% recycled glass (post-consumer or window shards in any proportion) regardless of the glass colour as well as 2% gypsum, limestone or silicon carbide (individually used). The heating process occurs at about 900°C in a conveyor belt furnace. The product has insulating, drainage, and load-bearing properties, is light weight, frost-proof, freeze-frost cycle resistant. The glass aggregate has the bulk density between 0.16-0.19 g·cm⁻³ and the compact material density is in the range of 0.21-0.25 g·cm⁻³. Heat conductivity is around 0.120 W·m⁻¹·K⁻¹ and water absorption has values between 6-10 vol. %. The compression strength is relatively high with values within the limits of 4.9-6.0 MPa. Recently, Misapor AG was focused only for the perimeter insulation products (Misapor AG, 2024; Misapor-Successful story, 2023).

Cellular glass aggregate manufactured by Glapor Werk Mitterteich is a light and insulating construction material prepared from 100% recycled glass waste. Its properties combine the physical and structural properties of glass with the insulation peculiarities of a usual closed-cell structure. According to (Glapor, 2017), Glapor insulation gravel is a load-bearing filling of expanded residual glass adequate for the use as a thermal insulation. Glapor filling gravel is a lightweight aggregate that can be utilized as a filling material. Suitable applications of Glapor products are: load-bearing heat insulation under foundation or floor slabs as well as perimeter insulation under floor construction. In accordance with (Glapor, 2017), bulk density values of Glapor products are

between 0.10-0.12 g·cm⁻³, heat conductivity has values lower than 0.12 W·m⁻¹·K⁻¹, and compression strength has values greater than 0.225 MPa.

According to the literature data, all industrial processes for manufacturing cellular glass aggregate is entirely based on conventional heating methods.

While the excellent potential of electromagnetic waves as energy carriers has been known since the mid-20th century, their application in industrial heating processes at high temperatures is still non-functional in the world. Only applications in transmissions and radars for over 70 years, while in the thermo-energetic domain there is information on their application only in drying processes and heating processes at low temperatures (Kharissova *et al.*, 2010).

In these circumstances, the Romanian company Daily Sourcing & Research initiated small-scale experimental research for manufacturing cellular glass aggregate using its own unconventional microwave heating technique. Several making recipes were tested in experiments conducted and published in the range 2019-2024.

Recipes containing coloured glass waste, calcium carbonate as an expanding agent, borax as a flux agent and water glass as a binder with important role in growing the mechanical strength (Ayadi *et al.*, 2010) were used in (Paunescu *et al.*, 2020a; Paunescu *et al.*, 2021). The process temperature was in the range of 830-855°C and the final product features included bulk density between 0.22-0.40 g·cm⁻³, heat conductivity in the range of 0.079-0.105 W·m⁻¹·K⁻¹, and compression strength within the limits of 5.9-9.5 MPa.

Other recipes experimentally used contained silicon carbide as an expanding agent mixed with glass waste (Paunescu *et al.*, 2022a; Paunescu *et al.*, 2020b) or silicon carbide mixed with borax and kaolin (Axinte *et al.*, 2021).

In other work (Paunescu *et al.*, 2023a), three manufacturing versions of cellular glass aggregate-microwave assisted were tried. In the first version, the mixture was composed of metakaolin, fly ash, alkaline earth alumina-silicate glass waste, and SiC as a foaming agent. The sintering/expanding temperature was 940°C. The second version included fly ash, blast furnace slag, alkaline earth glass waste, and SiC, the process temperature being 920°C. The third version had in the mix composition alkaline earth glass waste, glycerol as an expanding agent, and water glass. The temperature of this process was of only 850°C. Try results showed that bulk density was in the range of 0.21-0.26 g·cm⁻³, heat conductivity had values between 0.060-0.068 W·m⁻¹·K⁻¹, and compression strength varied between 5.0-8.5 MPa, its highest value being reached in the second version.

A making recipe including solid materials (glass waste, blast furnace slag, fly ash, borax, and zirconia) and liquid materials (glycerol and water glass) was used to prepare cellular glass aggregate in microwave field. Products had bulk density in the range of 0.29-0.38 g·cm⁻³, heat conductivity between 0.092-0.195 W·m⁻¹·K⁻¹, and compression resistance within the limits of 12.6-14.2 MPa (Paunescu *et al.*, 2022b).

The combination of two foaming agents, one liquid (0.9-1.2% glycerol) and one finely ground solid (0.9-1.1% calcium carbonate) was made in the paper (Paunescu *et al.*, 2023b) to test the experimental fabrication of a cellular glass aggregate from green and colourless glass waste. Water glass (between 3.7-4.2%) was also used in the liquid mixture. The temperature of the sintering/expanding process was between 835-840°C. The experiment results showed bulk density within the limits of 0.25-0.29 g·cm⁻³, heat conductivity in the range of 0.059-0.067 W·m⁻¹·K⁻¹, and compression strength between 7.1-7.6 MPa.

The current work aimed at testing other variants of the cellular glass aggregate composition, following to obtain a balanced correlation between its physico-thermal and strength performances. The excellent energy solution of predominantly direct using the microwave heating that has previously proven its validity on a small-scale was further chosen as the efficient heating method in this experiment.

2. Methods and Materials

The principal method of manufacturing cellular glass aggregate for insulation, load-bearing, and drainage applications in civil and industrial constructions as well as in road and bridge abutment constructions has been well-known on an industrial-scale for the last 50 years. The principle of manufacturing this product type is based on the preferential use of recycled glass waste from post-consumer drinking bottles and from the demolition and renovation of buildings. Finely ground, mixed with various expanding agent types and other additives in very small quantities, and sintered at temperatures imposed by the agent nature (generally, between 800-1000°C), the cellular aggregate is finally subjected to a fairly intense cooling to create internal tensions in its compact mass, thus allowing the detachment with relative ease of lumps with dimensions between 10-80 mm. This making method is very suitable to conveyor belt-tunnel kiln conditions, in which the raw material loaded onto the belt slowly travels a path that includes heating to the required temperature, annealing, and then relatively intense cooling contributing to the reception of cellular pieces falling off the conveyor belt at the cold end of the kiln.

The heating method adopted by all cellular glass aggregate manufacturers is conventional. This system has provided to Daily Sourcing & Research engineers a large area of research. Small-scale experiments performed in recent years have aimed to investigate the effect of high heating rates on the microstructural stability of cellular products. The first tests of direct microwave heating on glass-based materials showed an incompatibility between glass and microwaves, manifested by the destruction of the internal structure of materials subjected to this heating type. For this reason, the method of placing a protective screen made of microwave-absorbing materials (SiC and Si₃N₄) between the wave emitting source in one of the walls of the microwave oven and the sample

subjected to irradiation was adopted. The optimum thickness of the screen wall (which was a vertically placed cylindrical tube or a crucible) was experimentally determined to be 2.5 mm (Fig. 1a). In this way, the fully direct heating was turned into a mixed heating, in which predominantly, the electromagnetic waves penetrate the screen and come into direct contact with the sample, and partially, some waves are absorbed in the wall mass generating an intense heat transmitted to the material by thermal radiation. The main wave flux weakened in intensity so that direct heating no longer had a destructive character (Axinte *et al.*, 2019). The microwave emitting source was an 800 W-generator typical of microwave ovens used in household for food preparation. The oven adopted by the authors for the experiments was constructively and operationally adapted so that it could be used at very high temperatures of over 1100°C (Fig. 1b). According to previous works in the literature on the propagation of direct microwave heating (Jones *et al.*, 2002; Kitchen *et al.*, 2014), the thermal energy resulting from the contact of the waves with a solid material exhibits completely the opposite of that propagated in conventional processes. The waves, which are not a real energy source, but only energy carriers, activate their thermal potential in the core of the irradiated sample and there, the area of maximum energy intensity develops in its mass. After the heating is initiated, it volumetrically propagates from the inside to the outside, requiring intense thermal protection (with ceramic fiber resistant to 1200°C) of the screen placed between the source and the sample. Through this procedure, the microwave oven whose walls and vault are of sheet steel without other thermal protection system did not need insulation on its walls, even when the temperature of the heated material exceeded 1000°C, because the temperature measured on the outer metal surface did not exceed 70-75°C.



Fig. 1 – Experimental equipment
a – SiC/Si₃N₄-ceramic tube; b – adapted microwave oven.

In terms of chemistry, the two expanding agents react quite differently by heating the material mixture, but ultimately release gaseous compounds that contribute to forming gas bubbles and then, by cooling, create the cellular structure of the glass aggregate.

By decomposition, Na_2CO_3 releases CO_2 that remains blocked into the softened mass of raw material as well as Na_2O that enters in this composition playing the role of flux agent. The temperature at which Na_2CO_3 decomposition is initiated is about 550°C , the process being completed at over 900°C when Na_2O is completely obtained (Scarinci *et al.*, 2005).

Glycerol decomposes in the oxidizing environment of the furnace into several products from CO_2 to pure carbon and hydroxyl compounds. The process is early initiated at temperatures under 200°C and occurs up to about 850°C (Dou *et al.*, 2008). Being of carbonic origin, glycerol exhibits high affinity for oxygen. Thus, premature combustion of carbon can affect the expansion process due to losses of CO_2 (or CO) leaving the furnace enclosure. Therefore, the use of glycerol as a foaming agent is usually associated with the use of water glass (sodium silicate) to envelop the fine carbon particles.

Except for the two expanding agents mentioned above, the list of materials that make up the starting mixture contains predominantly green post-consumer drinking bottle, kaolin (powder), and water glass, to which is added water addition.

The recycled glass waste was washed, broken, ball milled and sieved, particle sizes below $80\ \mu\text{m}$ being selected for use in this experiment. The oxide composition of the waste was determined using the AXIOS X-ray fluorescence spectrometer from the Romanian Metallurgical Research Institute, with the following content: 71.8% SiO_2 , 1.9% Al_2O_3 , 11.8% CaO , 1.2% MgO , 13.1% Na_2O , 0.1% K_2O , 0.1% Cr_2O_3 (Dragoescu *et al.*, 2018).

The kaolin in powder state was commercially purchased with the grain size under $10\ \mu\text{m}$. Its chemical composition included: 57.6% SiO_2 , 37.8% Al_2O_3 , 0.4% CaO , 0.9% Fe_2O_3 , 0.6% MgO , 1.8% K_2O , and 0.3% P_2O_5 .

Water glass (or sodium silicate) was also purchased on the market in aqueous solution state with 38% concentration.

In this experiment, four experimental versions were adopted to produce cellular glass aggregate. The composition of these versions is shown in Table 1.

Table 1
Composition of the experimental versions (wt. %)

Composition	Version 1	Version 2	Version 3	Version 4
Green glass waste	92.3	81.0	89.4	86.7
Sodium carbonate	2.4	3.2	4.0	5.8
Kaolin	0.3	0.3	0.3	0.3
Glycerol	1.0	1.0	1.1	1.2
Water glass	4.0	4.5	5.2	6.0
Water addition	14.0	14.0	14.0	14.0

In conformity with the data in Table 1, except for kaolin and water addition values kept constant, values of other composition materials were modified as following: sodium carbonate increased from 2.4 to 5.8 wt. %, glycerol has grown from 1.0 to 1.2 wt. %, and water glass increased from 4.0 to 6.0 wt. % reaching maximum proportion values in the case of version 4.

Measuring the physical, mechanical, thermal, and microstructural features of the four cellular glass aggregate was achieved utilizing common analysis procedures. The bulk density was determined by the gravimetric method (Manual, 1999) and the porosity was identified by comparing method of the true and apparent density (Anovitz and Cole, 2005). The standard used for measuring the density and porosity is ISO 18754:2020. The heat conductivity was measured by the heat-flow method (ASTM E1225-04) and the compression resistance was identified with a TA.XTplusC Texture Analyzer (ASTM C552-17). The water uptake was measured through water-immersion method (ASTM D570). The microstructural appearance of cellular glass samples was examined with ASONA 100X Zoom Smartphone Digital Microscope.

3. Results and Discussion

The main operational parameters of the cellular glass aggregate making process are shown in Table 2. Heating the specimens was performed by the technique mentioned above of using the mixed heating, predominantly direct and partially indirect due to the placement of SiC and Si₃N₄-ceramic tube protected on its outer surface with high heat resistance. The thick layer (25-30 mm) does not constitute an obstacle in the path of electromagnetic waves emitted through the waveguide placed in the oven wall, because it is not a microwave susceptible material.

Table 2
Making process parameters of cellular glass aggregate

Parameter	Version 1	Version 2	Version 3	Version 4
Dry raw material/cellular glass aggregate amount (g)	450/ 436	450/ 437	450/ 436	450/ 436
Process temperature (°C)	832	835	839	845
Heating time (min)	33	34	36	39
Heating rate (°C·min ⁻¹)	24.61	23.97	22.75	21.15
Cooling rate (°C·min ⁻¹)	5.3	5.4	5.2	5.3
Specific energy consumption (kWh·kg ⁻¹)	0.79	0/81	0.86	0.93

The analysis of data presented in Table 2 showed that the required temperature for completing the sintering/expanding process is within the limits of 832-845°C. Using the unconventional heating method, the process duration was in the range of 33-39 min, indicating average heating rates between 21.15-24.61°C·min⁻¹, that significantly exceed the rates recommended in the literature (Scarinci *et al.*, 2005) in this type of processes (around 10°C·min⁻¹). The use of these very high heating rates led to the energy efficiency of the sintering/expanding process, the specific energy consumption obtained even in small-scale production conditions being remarkably low (between 0.79-0.91 kWh·kg⁻¹). The average cooling rate applied in this experiment had not higher values compared to that commonly used in the case of making known foam glass products, because the detachment of lumps from the compact mass of the processed material was not necessary.

Images of the four experimentally made cellular glass aggregate specimens are shown in Fig. 1.

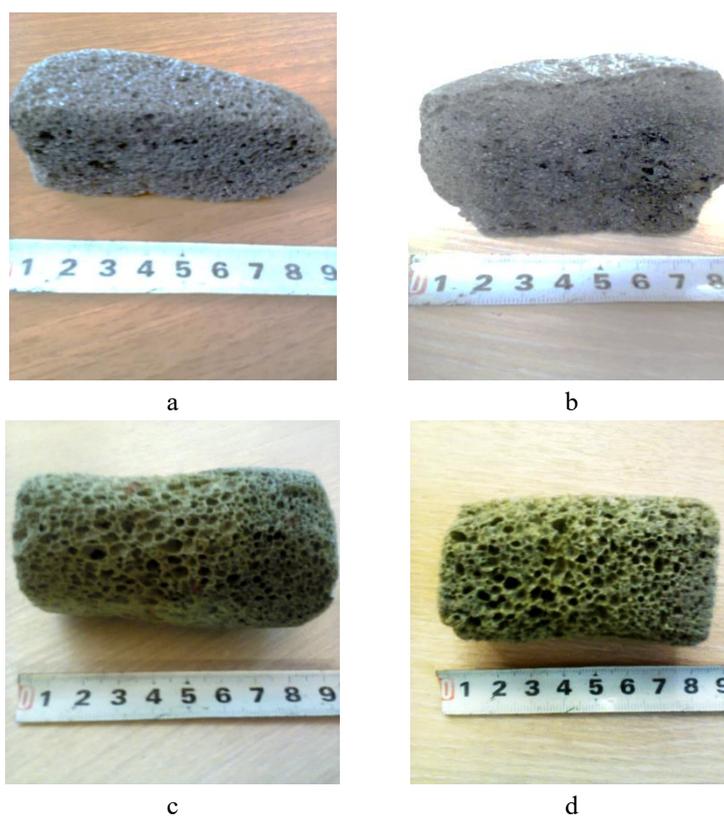


Fig. 1 – Images of cellular glass aggregate specimens
a – version 1; b – version 2; c – version 3; d – version 4.

Despite the very high heating rate values, no damage was observed at the macrostructural level of specimens.

The investigation of physical, mechanical, thermal, and microstructural characteristics of cellular glass aggregate specimens were performed according to the methods mentioned above. Results of these investigations are presented in Table 3.

Table 3
Characteristics of cellular glass aggregate specimens

Characteristic	Version 1	Version 2	Version 3	Version 4
Bulk density ($\text{g}\cdot\text{cm}^{-3}$)	0.25	0.23	0.22	0.20
Porosity (%)	84.8	85.1	85.7	86.1
Heat conductivity ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$)	0.070	0.065	0.062	0.058
Compression strength (MPa)	7.1	7.0	6.9	6.7
Water uptake (vol. %)	3.0	3.0	2.8	2.7
Pore size (mm)	0.1-0.5	0.4-0.9	0.7-1.7	1.0-2.4

For identifying peculiarities at microscopic level of cellular glass aggregate specimens, images obtained within the ASONA Smartphone Digital Microscope were examined. The pictures are exposed in Fig. 2.

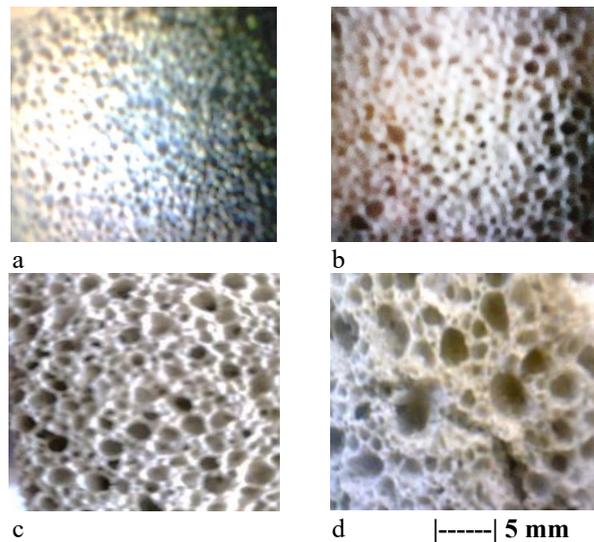


Fig. 2 – Microstructural aspect of cellular glass aggregate specimens
a – version 1; b – version 2; c – version 3; d – version 4.

The pictures above show a very homogeneous organization of cells in the matrix of the cellular glass aggregate samples, especially in the case of versions 1-3. In the case of sample (d) made in version 4, a tendency towards a coarser microstructure can be observed, the appearance and dimensions of the cells varying to a greater extent compared to the microstructures of the other specimens. On the other hand, there is no tendency towards coalescence of neighboring cells, all cells being closed. The ranges of cell size variation are shown in Table 3. Obviously, the smallest cells compose the microstructure of sample (a) made in version 1. The cell sizes increase slightly in the case of samples (b) and (c), reaching that in the case of sample (d)-version 4, the range of sizes to be 1.0-2.4 mm.

Given that cellular glass aggregate (or cellular glass gravel) has been an industrially manufactured product in the world for several decades, the current work aimed at applying innovative solutions on a small-scale.

The most important of them is obviously the replacement of traditional conventional methods for heating the raw material with the microwave irradiation technique through an own version of direct unconventional heating combined with indirect conventional heating (through thermal radiation). The energy efficiency of this technical version was realized through the possibility of consistently increasing the heating rate value without affecting the structural integrity of the expanded product.

The other solution tested in this work was a technological one by combining solid and liquid foaming agents. While the liquid agent (glycerol) is interesting for some manufacturers, its combination with sodium carbonate has not been tried yet. By comparison with calcium carbonate used frequently in expansion processes, sodium carbonate has a much lower ability to expand the glass, but through decomposition it releases, except for CO_2 , Na_2O that enters into the raw material composition and is one of the most important fluxing agents.

Of the four experimental versions presented in the paper, the one made by combining 4 wt. % sodium carbonate with 1.1 wt. % glycerol and the addition of 5.2 wt. % water glass and 0.3 wt. % kaolin (i.e. version 3) was chosen as the optimal one. This had the most adequate features (bulk density of $0.22 \text{ g}\cdot\text{cm}^{-3}$, heat conductivity of $0.062 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, and compression resistance of 6.9 MPa), including also a homogeneous microstructure with closed cells having dimensions between 0.7-1.7 mm.

4. Conclusions

The objective of this work was to contribute to the improvement of the cellular glass aggregate manufacturing process, especially in energy terms, by implementing the unconventional technique of microwave heating. Although the tests were carried out on a small-scale with important differences compared to the particularities of the industrial process, the use of electromagnetic waves

proved its energetic viability and the very high heating rates did not affect the quality of the final product. Cellular glass aggregates made under these conditions had excellent heat insulation properties as well as high mechanical strength values, being at the level of similar materials industrially produced.

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UTILIZAREA COMBINATĂ A AGENȚILOR DE SPUMARE
SOLID ȘI LICHID PENTRU FABRICAREA AGREGATULUI DE STICLĂ
CELULARĂ ASISTAT DE MICROUND

(Rezumat)

Agregatul din sticlă celulară cu excelente proprietăți termoizolante și rezistență mecanică a fost proiectat și testat în condițiile utilizării metodei neconvenționale de încălzire cu microunde. Carbonatul de sodiu și glicerolul au fost utilizați concomitent ca agenți de spumare. Sticla verde de băutură post consum a fost materia primă predominantă în amestecul care mai conține caolin și apă de sticlă. Produsele celulare au avut densitatea în vrac între 0,20-0,25 g·cm⁻¹, conductivitatea termică în intervalul 0,058-0,070 W·m⁻¹·K⁻¹ și rezistența la compresiune în limitele 6,7-7,1 MPa. Eficiența energetică a procesului a fost remarcabilă, consumul specific de energie fiind între 0,79-0,93 kWh·kg⁻¹.